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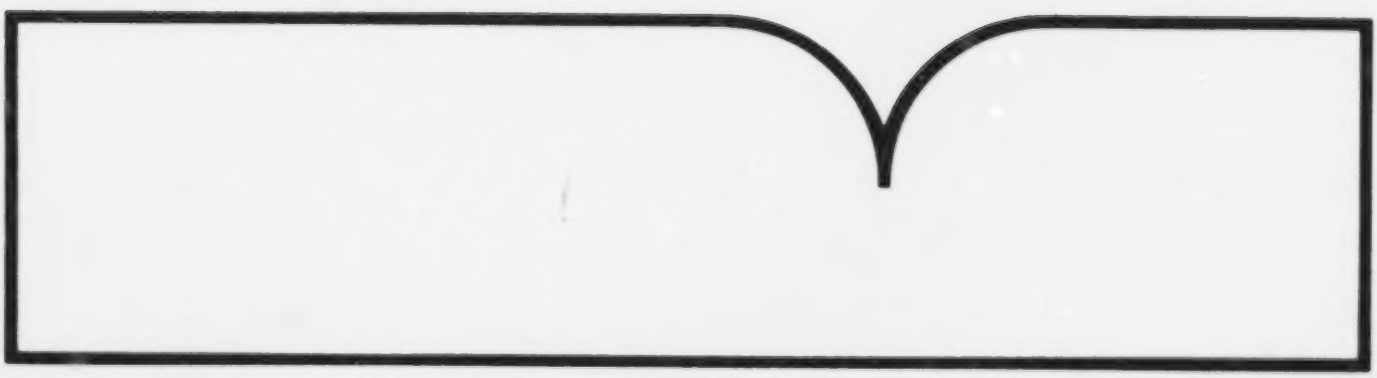

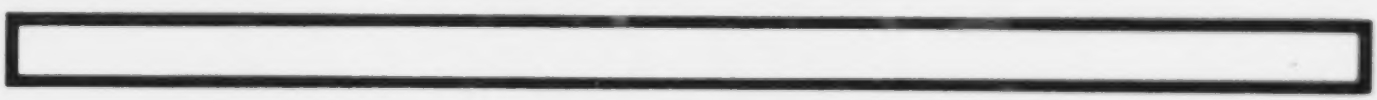

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BACKGROUND DOCUMENT

RESOURCE CONSERVATION AND RECOVERY ACT

SUBTITLE C - HAZARDOUS WASTE MANAGEMENT SYSTEM

SECTION 3001

INDENTIFICATION AND LISTING OF HAZARDOUS WASTE

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

(TCLP)

U.S. ENVIRONMENTAL PROTECTION AGENCY

OFFICE OF SOLID WASTE

March 10, 1986

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This document provides data and detailed information on the development and evaluation of a second generation leaching procedure, known as Toxicity Characteristic Leaching Procedure (TCLP).
This method is intended to serve as an indicator of waste; if mismanaged, has the potential to pose a significant hazard to human health or the Environment due to its ability to leach significant concentration of Toxic Concentrations.

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BACKGROUND DOCUMENT:

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

I. INTRODUCTION

Under Section 3001 of tne Resource Conservation and Recovery Act (RCRA), EPA was charged with identifying those wastes which pose a hazard to human health and the environment if improperly managed. In carrying out this mandate, EPA identified a number of characteristics which, if exhibited by a waste, would indicate that the waste is a hazardous waste and should be managed under Subtitle C of RCRA. One of these characteristics, the Extraction Procedure (EP) Toxicity Characteristic (EPTC) (40 CFR 261.24), was intended to identify wastes which pose a hazard due to their potential to leach significant concentrations of specific toxic species.

The EPTC entails the use of a leaching test, the Extraction Procedure (EP), which is used in determining if an unacceptably high level of ground water contamination might result from improper waste management. At the time of promulgation of the EPTC, however, EPA recognized one major drawback of the EP. Specifically, the EP was optimized for evaluating the leaching of elemental rather than organic contaminants, and was thus limited in its application to organic contaminants (U.S. EPA, 1980).

In addition to addressing the leaching of organics, the Agency felt that other aspects of the EP could be improved. For example, the EP protocol is known to suffer a number of operational difficulties (detailed in Section III of this

background document) that, while not critical, warrant attention.

Congress also recognized the shortcomings of the EP, and amended RCRA in 1984, directing EPA to make changes in the EP to insure that it accurately predicts leaching potential.

This background document provides data and detailed information on the development and evaluation of a second generation leaching procedure, known as the Toxicity Characteristic Leaching Procedure (TCLP).

II. DEVELOPMENT OF THE TCLP

A. Objectives

EPA's intent, then, was to develop an improved leaching test method suitable for use in evaluating wastes containing organic toxicants. The method is intended to serve as an indicator of when a waste, if mismanaged, has the potential to pose a significant hazard to human health or the environment due to its ability to leach significant concentrations of toxic contaminants.

When the EP was developed, the Agency had little empirical data upon which to base its assumptions regarding accuracy (U.S. EPA, 1980). Hence, while the little data that was available regarding accuracy was used in developing the EP, it was primarily based on what was reasonable, as well as what would provide a reproducible (precise) test protocol. While improved reproducibility is one objective of the TCLP, the major objective was to accurately model the mobility of constituents, particularly organic constituents.

Organic constituents were of particular concern because the

Agency believes that the EP was, and still is, adequate in its accuracy with respect to identifying wastes that have the potential to leach significant concentrations of the elemental contaminants. The research conducted during development of the TCLP largely confirmed this. For the most part, when a waste was demonstrated to leach significant concentrations of the elemental toxicants in the field studies (described in further sections of this background document), high concentrations of these elements were also noted in EP extracts (Francis et al., 1984; Francis et al., Feb., 1986; TAI, 1985).

Other objectives of the TCLP were that it be relatively inexpensive to conduct, that, if possible, it yield an extract amenable to evaluation with biological toxicity tests, and that it also model the mobility of inorganic species. This last objective would permit EPA to be able to evaluate the mobiltiy of both organic and inorganic contaminants with one test.

B. Disposal Environment and Model

The EP was designed to simulate the leaching that would result when a solid waste is codisposed with municipal waste in a sanitary landfill. It was intended to be a first order approximation of the leaching action of the low molecular weight carboxylic acids generated in an actively decomposing sanitary landfill. Acetic acid, one of the more prevalent carboxylic acids in municipal waste leachate, is added to deionized distilled water to make up the extracting medium used in the EP. The acetic acid models primarily the leaching of metals.

The sanitary landfill codisposal model was selected as

a reasonable worst case mismanagement scenario for the EP because the Agency was concerned that potentially hazardous waste, if not brought under RCRA control, may be sent to sanitary landfills, with a resulting high degree of leaching activity (U.S. EPA, 1980). In determining what form of mismanagement to model in developing the TCLP, the Agency considered several alternatives. These included sanitary landfill codisposal, segregated management, codisposal with industrial waste in a non-hazardous waste (Subtitle D) landfill, and codisposal with hazardous waste in a hazardous waste (Subtitle C) landfill which suffers some sort of system failure.

As with the EP, the scenario selected for the TCLP is co-disposal with municipal waste in a sanitary landfill. Hence, the concern that potentially hazardous waste, if not brought under the control of the RCRA hazardous waste system, may be sent to sanitary landfills and result in a high degree of leaching, has not changed. A recent survey conducted for the Office of Solid Waste (JRB, 1985), concluded that "...in general, Subtitle D landfills accept industrial wastes but not organic solvents or liquids. While the Agency does recognize that fewer industrial solid waste are being disposed of in this manner as compared to a few years ago, the Agency believes that this scenario represents a reasonable form of mismanagement with respect to potential for causing ground water contamination. Because Subtitle D landfills generally do not accept substantial amounts of organic solvents, the Agency has retained the concept that the extraction fluid be derived from the decomposition of sanitary refuse.

EPA believes that industrial facilities dedicated to the management of only one waste, or the waste from only one generator, are likely to pose less of a hazard than would general sanitary landfills, since the design and operation problems are simpler and the operator has much more information on the properties of the wastes before and while the facility is in operation. With respect to modeling a Subtitle C landfill, since such facilities must comply with design, operation, and monitoring provisions of the Section 3004 regulations, EPA believes that the likelihood of wastes disposed of in such facilities causing ground water contamination is much lower than if the waste was placed in a Subtitle D facility which lacks these forms of secondary protection.

Hence, the experiments used to develop the TCLP were set up to conform as closely as possible with the codisposal model used for the EP. The intent was to model the early stages of landfill decomposition (generally the first few years of landfill life), as it is during this stage that the leachate would be most aggressive in its mobilizing ability, both in terms of leachate acidity and in complexation ability. Specific features of the model were that the landfill is composed of 5 percent industrial solid waste and 95 percent municipal waste, and that the character of the leaching fluid that the waste would be exposed to is a function of the decomposing refuse in the landfill (U.S. EPA, 1980). In developing the TCLP, the model and assumptions used in developing the EP have been retained.

C. Experimental Design

EPA, through an interagency agreement with the U.S. Department

of Energy's Oak Ridge National Laboratory (ORNL), has conducted a research program designed to develop the TCLP. The TCLP development program was split up into three phases. Phases I and II, and part of Phase III have been completed. Phase I consisted of an initial data gathering effort in which a number of wastes were leached with a leachate derived from municipal refuse. The wastes were also extracted with a variety of laboratory leaching media and contact procedures. Phase I was designed to narrow the universe of potential candidate leaching procedures. In Phase II, additional wastes were leached and the candidate procedures refined into the draft TCLP. Following this phase of testing, public assistance and review of the draft was solicited. Phase III involves evaluation of the TCLP as to its intra- and inter-laboratory precision and ruggedness.

The overall approach employed in Phase I was as follows:

1) Large-scale field lysimeters were filled with domestic and commercial refuse and used to generate a municipal waste leachate (MWL).
2) The MWL was used to leach four industrial solid wastes in large columns.
3) The leachate concentrations of a number of organic and inorganic species that were present in each waste were measured over time.
4) A total of 34 laboratory leaching tests were run on the four wastes to assess their accuracy in modeling the results of the lysimeter/column experiments. These tests included both

column and batch procedures using four leaching media (i.e., sodium acetate buffer, carbonic acid, water, and actual municipal waste leachate), and four media to waste ratios (i.e., 2.5, 5, 10, and 20 to 1). In addition, the EP and a sequential batch leaching procedure were also investigated.

5) Target Concentrations (TC), were established for each constituent based on the lysimeter/column leaching curves, by calculating the amount of constituent leached over a specific leaching interval (i.e., an amount of leachate equal to twenty times the weight of the original industrial solid waste - twenty to one liquid to solid ratio).

6) Laboratory leaching test results were compared to the TCs, and the two laboratory tests that best replicated lysimeter results were selected for further evaluation in Phase II.

Phase II of the program involved extensive evaluation and verification of Phase I:

1) Seven wastes were leached in essentially the same experimental arrangement as used in Phase I.

2) Each waste was subjected to the two "best" leaching procedures selected from Phase I, as well as the EP.

3) The single procedure which best satisfied the objectives presented earlier in this background document was selected as the draft TCLP.

4) The draft TCLP was then circulated to interested members of industry, academia, environmental groups, and others with interest and experience in conducting such tests, for comment.

Phase III of the program involved subjecting the resulting TCLP

to an evaluation of ruggedness and precision. This work has been partially completed and the design and results to date are summarized in this background document. Another part of Phase III which is also in progress is a multi-laboratory collaborative evaluation of the draft TCLP. (The TCLP has evolved to its present form in response to both Agency activities as well as to comments received on the circulated draft). The following sections present the experimental program and the results.

D. Phase I Results

The ORNL Phase I report explains in detail the experimental approach and describes the results obtained during the first phase of testing (Francis et al., 1984). Briefly, lysimeter leachate target concentrations were established based on both practical considerations and the need to represent a mid-to-long term leaching interval or exposure period. This was important as the purpose of the leaching test is primarily to evaluate the migratory potential of chronically toxic contaminants. (The rationale for the determination of leachate target concentrations is presented in Kimmell and Friedman, 1984).

The various laboratory procedures tested were then compared as to their ability to reproduce the lysimeter leachate target concentrations. The absolute value of the percentage difference for each target concentration/leaching test concentration pair was determined, averaged for each leaching procedure, and then each procedure was ranked from the lowest to highest difference and evaluated for significance using Duncan's multiple range test. These analyses identified most of the laboratory procedures

as being equally predictive of lysimeter leachate target concentrations, particularly where the organics were concerned (Francis et al., 1984).

No single procedure will be able to accurately predict leachate concentrations for all compounds in all waste matrices. EPA therefore picked the procedures which seemed to most closely model lysimeter leachate target concentrations using the absolute value of the percentage difference. Factors other than average percentage difference, such as ease and expense of operation, applicability to both organics and inorganics, and applicability to biological testing, were also taken into account. These factors were identified previously in this background document as objectives for the TCLP.

On the basis of all these considerations, two procedures, similar in concept and operation to the current EP, were selected for further work in Phase II. Both of these procedures use a 20:1 liquid to solid ratio (i.e., an amount of extraction fluid equal to twenty times the weight of the solid phase of the waste) and involve a batch-type extraction. One procedure uses a 0.1 N pH 5 sodium acetate buffer solution as the extraction medium, and the other uses CO_2 saturated deionized distilled water (i.e., carbonic acid). The rationale for the selection of these two procedures is laid out in Kimmell and Friedman, 1984.

E. Peer Reviews

A number of peer reviews were conducted at various stages of the TCLP development program. The general tone of these reviews was always positive, and all of the suggestions of

the peer reviewers were given full consideration.

One such review which had profound effect on the way the TCLP development data was analyzed, is a review conducted by the Agency's Science Advisory Board (SAB, 1984). At the end of Phase I, the Environmental Engineering Committee of the Science Advisory Board (SAB) was asked by EPA to review and provide recommendations concerning the development program and the selected methods (U.S. EPA, Feb 2, 1984). Overall, the SAB found that the experimental approach taken reasonably represented an actual landfill. The SAB did, however, question the statistical methodology used to evaluate the Phase I data and recommended that the data be re-evaluated using additional statistical analyses. Their primary concerns were the need to provide more resolution in the data through the use of more powerful statistical tests, the need to indicate the direction of the statistical differences (i.e., were individual laboratory tests generally more or less aggressive than lysimeter targets), and the need to examine the data for possible compound or class related trends.

The SAB review resulted in a number of interrelated actions. First, the Research Triangle Institute, under contract to EPA's Statistical Policy Branch, conducted an independent statistical review of the methods used to evaluate the Phase I data (RTI, August 10, 1984). Concurrently, statisticians from ORNL reviewed the concerns of the SAB and prepared a response (Francis, September 7, 1984). Finally, ORNL examined the RTI comments in light of their own, the result of which was the application of a number of tests to the ORNL data (Francis, October 26, 1984 and

Francis et al., Feb., 1986), on both Phase I and II of the research. The results of these statistical analyses are summarized in the next section of this background document.

In May of 1984, the SAB also received comments on the ORNL research from Environment Canada's Wastewater Technology Center (Cote, May 11, 1984). EPA responded to these comments in it's response to the comments of the SAB (U.S. EPA, March 14, 1985), and provided more information to the SAB in a subsequent submission (U.S. EPA, May 9, 1985).

In addition to the review conducted by the SAB, two formal peer-reviews were conducted on the research at the ORNL. The first of these reviews took place on October 30, 1981, and involved three reviewers. This review was conducted during the planning stage for the ORNL research and dealt mainly with the technical approach to the experiments that were to be conducted at the ORNL. Written comments were received from two of the reviewers (Helling, November 16, 1981 and Brunner, December 17, 1981).

The second peer-review took place at the end of February, 1983, at a point where most of the data on the first phase of the ORNL research was available. This review centered on the examination of a number of approaches to the establishing of lysimeter leachate target concentrations, and approaches to data analysis. Again, three peer reviewers were invited to participate. Two of the reviewers provided written comments (Helling, March 9, 1983 and Anderson, March 17, 1983). The approaches to establishing target concentrations and data analysis are discussed fully in the ORNL final reports (Francis et al., 1984 and Francis et al.,

Feb., 1986).

F. Phase II and Combined Phase I and II Results

As a result of the SAB comments and ORNL's efforts to evaluate the TCLP development data using the best possible statistical techniques, a number of additional statistical tests were applied to both the Phase I and Phase II data individually, and the Phases I and II combined data (Francis et al., Feb., 1986). In addition, EPA subjected the ORNL data to an independent statistical analysis (TAI, 1985).

Before summarizing the results of these statistical analyses, it is important to bear in mind that no single leaching procedure will be able to accurately predict leachate concentrations for all compounds in all waste matrices. The idea was to select the procedure which most consistently modeled the field lysimeters. Another consideration was the need to minimize the occurrence of false negative results (i.e., the situation where the leaching test falsely identifies the waste as non-hazardous - in this case the leaching test would be less aggressive than field results). While it is important to also minimize the occurrence of false positives, EPA believes that minimizing false negatives is more important, since the consequences of false negative results are more environmentally serious. In addition, other factors, such as ease and expense of operation, applicability to both organics and inorganics, reproducibility, and applicability to biological testing (the original objectives in developing the TCLP), were also considered in selecting the most appropriate leaching medium.

Table 1 summarizes the results of four of the more

important statistical analyses applied to the data comparing lysimeter to laboratory results. This table presents comparisons between three extraction media (i.e., acetate buffer, carbonic acid and EP leaching medium), and includes the results for both organics and inorganics from both phases of testing. Only statistically significant results are presented here. A different letter indicates a statistically significant difference at the 5 percent level, with all extractions having an "A" value being closest to the lysimeter results. The results reported in this table come from several references. The Phase I and II actual and absolute percent differences come from TAI, 1985. These analyses were also done by ORNL (Francis et al., 1984 and Francis et al., Feb., 1986), and the results compare well. The Phase I multivariate and precision analyses come from Francis et al., Mar. 14, 1985. The Phase II and combined Phase I and II multivariate and precision analyses come from Francis et al., Feb., 1986. Note that these references present the actual numerical results of the statistical tests.

The first test, the absolute percent difference, gives a crude indication of accuracy of each of the methods. Looking at Table 1, the most apparent conclusion is that there is essentially no significant differences among the leaching media as to their ability to duplicate field results. This is especially true for organic compounds. A better means of indicating significant differences in accuracy is believed to be the multivariate analysis, the results of which are also presented. This test indicates that the acetate buffer extraction is significantly more accurate than the other media. In carrying out the multivariate

Table 1: Summary Table

Statistical Analysis of the data used to
Develop the Toxicity Characteristic Leaching Procedure
Statistical Significance Only [1]

	INORGANICS			ORGANICS			INORGANICS AND ORGANICS		
	Phase I	Phase II	Phase I+II	Phase I	Phase II	Phase I+II	Phase I	Phase II	Phase I+II
ABSOLUTE % D [2]									
ACETATE	B	A	A	A	A	A	A	A	A
CARBONIC	A	A	A	A	A	A	A	A	A
EP	B	A	A	A	A	A	A	A	A
ACTUAL % D [3]									
ACETATE	-B	-AB	-B	+A	+A	+A	-A	+A	-A
CARBONIC	-A	+B	+A	+A	+A	+AB	-A	+B	+B
EP	-AB	-A	-B	+A	+A	+B	-A	+A	+AB
MULTIVARIATE [4]									
ACETATE							A	A	A
CARBONIC							A	B	B
EP							B	B	B
PRECISION (C.V.) [5]									
ACETATE	A	A	A	D	B	B	B	B	B
CARBONIC	A	B	B	B	C	C	A	C	C
EP	ND	B	B	ND	C	C	ND	B	B

1. A different letter indicates statistical significance at the 5% level, an "A" value being closest to the lysimeter target values.
2. Absolute value of the percentage difference between laboratory concentration and the lysimeter field results.
3. Actual value of the percentage difference between laboratory concentration and the lysimeter field results. A negative value indicates the laboratory concentrations to be higher than lysimeter field results.
4. Ranks based on the Mahalanobis distance (See Francis et al., 1985).
5. Precision, as coefficient of variation categorized as follows. A=0-19, B=20-39, C=40-59, D=60+, ND = Not Duplicated.

analysis, the results for organics and inorganics were not examined separately (See Francis et al., Feb., 1986).

The actual percent difference, while also giving an estimate of accuracy, can be used to estimate the aggressiveness of the leaching media relative to the field results. A negative value indicates that the extraction is more aggressive than the lysimeter field model. Looking at Table 1, once again there are few significant differences among the leaching media as to their ability to extract organic compounds. All the values for the organics comparisons are positive values, indicating that the laboratory tests are generally less aggressive than the lysimeter model. This is most probably a function of the wastes being extracted in a device which does not fully prevent loss of volatiles (i.e., losses of volatile organics to the headspace in the extraction vessel), and also a function of the fact that the field leachates were analyzed unfiltered, whereas the laboratory extracts were analyzed filtered. Higher concentrations of some organic compounds, especially polyaromatic hydrocarbons, were observed in the unfiltered extracts.

For inorganics, the actual percent differences test did produce some negative values, indicating that some of the leaching media tested were more aggressive towards inorganics than the lysimeter field model. In Phase I, this was true for all three leaching media. In Phase II, however, this was only true for the acetate buffer and EP leaching media. Hence, in Phase II, the carbonic acid leaching media was generally less aggressive towards inorganics than the lysimeter field model (Francis et al., Feb.,

1986 and TAI, May, 1985).

Although the Phase I data indicated that the carbonic acid leaching medium most closely approximated the lysimeter results (Francis et al., 1984), when the Phase II data were taken into account, the sodium acetate buffer leaching medium seemed to be the most appropriate (Francis et al., Feb., 1986 and TAI, May, 1985). Given the most weight in this evaluation was the multivariate analysis.

The analysis of precision, the last statistical analysis presented in Table 1, also indicated that the acetate buffer extraction would provide a more precise test procedure than either of the other two media. (The precision and ruggedness of the TCLP are further discussed in a subsequent section of this background document). In addition, the acetate buffer system offers a number of operational advantages over either the carbonic acid medium or the EP leaching medium. Finally, use of the acetate buffer system should minimize the occurrence of false negative results, since the Phase II inorganics analyses indicated that the carbonic acid medium was less aggressive than the lysimeter field results.

For the above reasons, the sodium acetate buffer system has been selected as the medium of choice. Perhaps the only objective that may have been compramised by selection of the acetate buffer system, was the objective to have a leaching medium that is applicable to biological testing. Although the acetate buffer system will complicate biological testing, it should not preclude bioassay evaluation of TCLP extracts entirely.

Note that the TCLP procedure involves the use of a dual leaching media, with the use of an acetic acid solution (rather than a buffer solution) for certain wastes that are significantly alkaline in nature. The rationale for this action is presented in a subsequent section of this background document.

Phase III of the TCLP development program involves an evaluation of ruggedness and precision as well as a multi-laboratory collaborative study. Since the design of these studies, and hence the results, are a function of how EPA addressed some of the operational and other aspects of the TCLP, a discussion of Phase III is presented following the section of this background document which discusses these other aspects of the TCLP.

G. Accuracy of the TCLP

In an effort to quantify how well the TCLP compares to the field model, the distributions of the actual and absolute percent differences between concentrations observed in the field model and those observed in the acetate buffer system chosen for the TCLP have been examined (TAI, 1985). Results of these comparisons indicate that roughly half of the 95 individual target compounds (from the 11 wastes examined in both Phases I and II), were within -32 percent and +76 percent of their respective field lysimeter target concentrations. Roughly three fourths of the 95 individual target compounds were within -80 percent and +86 percent of their respective field lysimeter target concentrations. The standard deviation of the total distribution (which is skewed) in this case is 182 percent (TAI, 1985).

These numbers indicate that the acetate buffer system duplicates

the field lysimeter target concentrations for approximately three fourths of the target compounds within one standard deviation of the distribution. This is particularly significant since the laboratory test duration is 18 hours, whereas the field lysimeters were run for approximately three months. EPA believes that the accuracy of the TCLP is adequate in terms of indicating the potential for wastes to pose a hazard if mismanaged.

As an additional matter, the Agency believes that the predicted degree of contaminant concentration in leachate could reasonably occur in the course of other types of land based management (e.g., surface impoundments). The TCLP, as well as the EP, basically involve mixing the waste with an aqueous leaching media, and seeing if toxic contaminants can migrate from the waste to a significant degree. If such mobility is demonstrated, EPA believes that the waste in question poses a potential hazard to ground water, and that proper management controls need to be instituted to preclude unacceptable contamination of the ground water. This applies to the leaching of both organics and inorganics.

First, as the leaching experiments supporting the development of the TCLP demonstrated, minor changes to primarily aqueous media do not generally affect the leaching of organic compounds. This same phenomenon has been observed in other research efforts (McKown et al., 1981 and Spellenberg, 1982). For inorganics, the acidity afforded by the TCLP leaching fluid accounts for the possibility that wastes could be subjected to mild acidic conditions occuring in other types of land disposal environments.

Of course, wastes do have the potential to be subjected to more aggressive conditions that might be better modeled through the use of strong inorganic acids, alkalies, or solvents. However, the Agency believes that such conditions do not have to be modeled by the TCLP, since the Agency is taking steps to ban the disposal of solvents and corrosives in land-based management facilities (See Land Disposal Restrictions Rule, proposed on Jan. 14, 1986, (51 FR 1602). The survey referred to earlier (JRB, 1985), found that even without the ban, Subtitle D facilities generally accept only small amounts of organic solvent waste (e.g., < 1-2 percent of the total waste accepted). In addition, EPA will consider listing specific wastes as hazardous, when their normal management or their potential for mismanagement dictates more aggressive conditions.

H. Additional Experimentation

Eleven wastes and 95 target compounds that leached from these wastes were examined in the TCLP development program. The amount of work involved here was substantial. EPA is aware, however, of one waste type, specifically those of moderate to high alkalinity, that was not adequately represented by the 11 wastes, and has made changes in the TCLP to insure that the potential environmental damage that may be caused by these wastes was not underestimated. (These changes are detailed in a subsequent section of this background document).

Although the argument can be made that EPA should examine more wastes in the ORNL lysimeters, EPA believes that further testing is unnecessary. While more testing might provide that much more of a basis for evaluating the accuracy of the TCLP, EPA believes

that it will not alter the conclusions that have already been reached. Perhaps the most significant of these is that the leaching of most organic compounds is unaffected by minor changes in primarily aqueous extraction media.

III. OPERATIONAL ASPECTS

As indicated previously, in moving from the EP to the TCLP protocol, the Agency hoped to improve the test procedure and eliminate some steps in the EP procedure which have caused difficulty for analysts. These include the need for continual pH adjustment, which is time consuming and serves as a source of imprecision, and the difficulty in performing the initial and final liquid/solid separations, which currently involves 0.45 um pressure filtration. In addition, the need to more precisely describe what constitutes acceptable agitation and to adequately prevent volatilization of organic compounds during extraction was critical. Finally, other minor changes in the EP protocol, such as shortening the duration of the test and accounting for the loss of waste materials to the sidewalls of sample containers, were felt to be of use in lowering the cost of the test and in improving its overall precision. Thus the Agency believes that development of a second generation extraction procedure was of value even if the EP was found to be acceptable for organic contaminants.

These operational aspects of the test procedure are discussed below. As an aid, Table 2 presents a comparison between the EP and the TCLP, in terms of procedural aspects. Figures 1 and 2 present the flow diagrams for each method,

spectively.

A. pH Adjustment

The EP procedure involves continual pH adjustment or titration of a waste/deionized distilled water slurry to a pH of 5.0 ± 0.2. The procedure calls for periodic pH adjustments if necessary, at 15 minute intervals for up to 6 hours or more. This is very tedious, time consuming and expensive, and is also probably the single most important element in the EP protocol contributing to variability. Using a buffer system eliminates the problem of pH adjustment since this medium does not require such adjustment during extraction.

B. Liquid/Solid Separation

The initial liquid/solid separation problems are due to the tendency for some materials, such as certain types of oily wastes, to clog the 0.45 um filter, and prevent filtration even if considerable pressure (75 psi) is applied. This problem is serious, since materials which do not pass the 0.45 um filter are treated as solids even if they physically appear to be liquid. These (liquid) wastes are then carried through the EP extraction as a solid.

This is particularly serious for oily wastes, since oils have been known to frequently migrate to ground waters. It is important for the liquid/solid separation to treat, as liquids, those materials which can behave as liquids in the environment. It is important to recognize, however, that some materials, such as many paint wastes, while they have some liquid properties, they will generally behave as solids in the environment (i.e., will

Table 2

Comparison of the Extraction Procedure (EP) and the Toxicity Characteristic Leaching Procedure (TCLP)*

Item	EP	TCLP
1) Leaching Media	- 0.5 N Acetic acid added to distilled deionized water to a pH of 5 with 400 ml maximum addition. - Continual pH adjustment.	- 0.1 N pH 2.9 acetic acid solution for moderate to high alkaline wastes and 0.1 N pH 4.9 acetate buffer for other wastes.
2) Liquid/solid separation	- 0.45 um Filtration to 75 psi in 10 psi increments. - Unspecified filter type.	- 0.6-0.8 um Glass fiber filter filtration to 50 psi.
3) Monolithic material/particle size reduction	- Use of Structural Integrity Procedure or grinding and milling.	- Grinding or milling only. - Structural Integrity Procedure not used.
4) Extraction Vessels	- Unspecified design. - Blade/stirrer vessel acceptable.	- Zero-headspace vessel reguired for volatiles. - Bottles used for non-volatiles. - Blade stirrer vessel not used.
5) Agitation	- Prose definition of acceptable agitation.	- Rotary agitation only in an end-over-end fashion at 30 $\pm$ 2 rpm.
6) Extraction time	- 24 hours.	- 18 hours.
7) Quality Control requirements	- Standard additions required. - One blank per sample batch.	- Standard additions required in some cases. - One blank per day or for every new batch of extract. - Analysis specific to analyte.

* All other attributes between the two tests are generally the same, although there are some minor differences. Note also that while the EP only addresses those species for which National Interim Primary Drinking Water Standards (NIPDWS) exist, the TCLP can be applied to other toxicants.

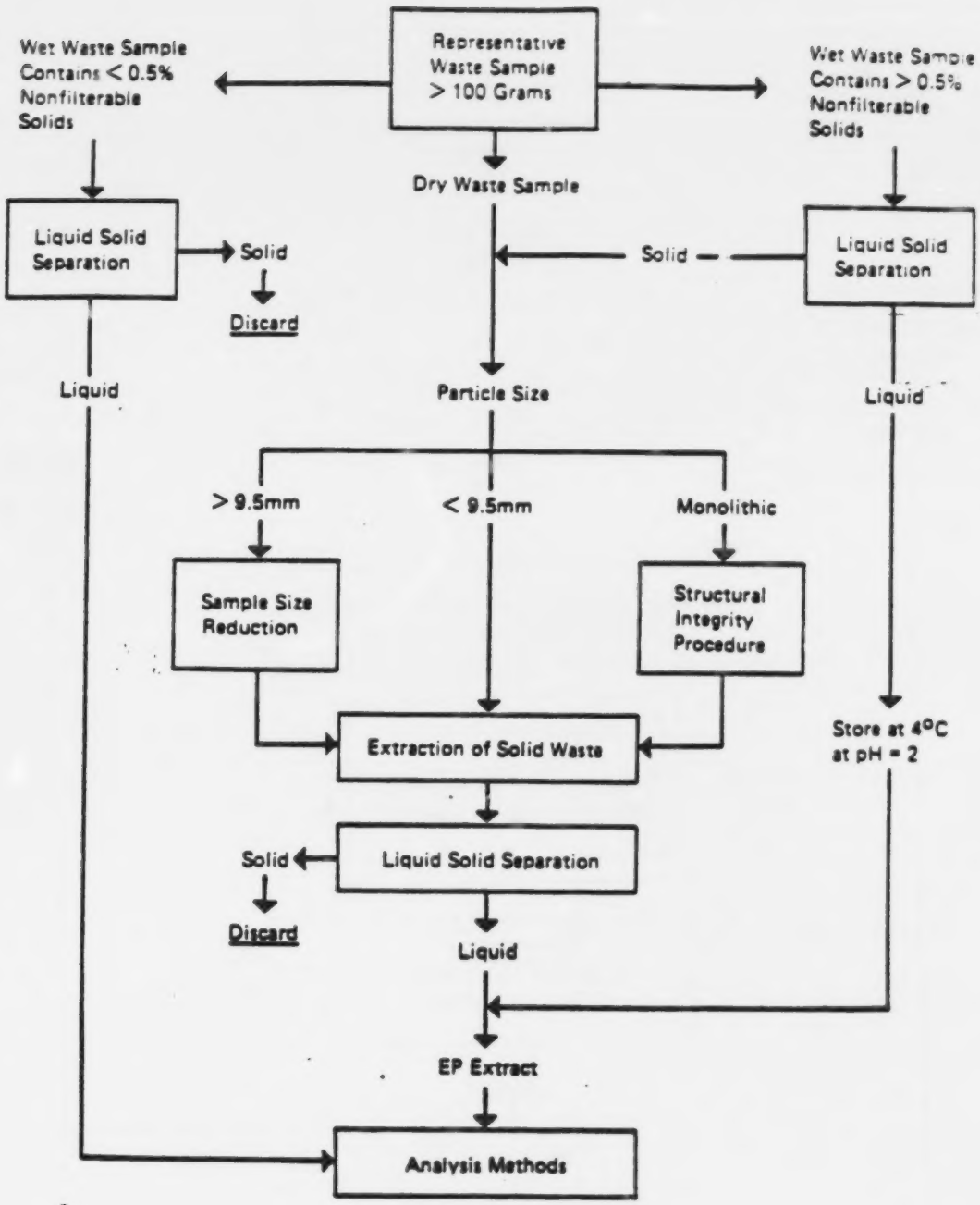
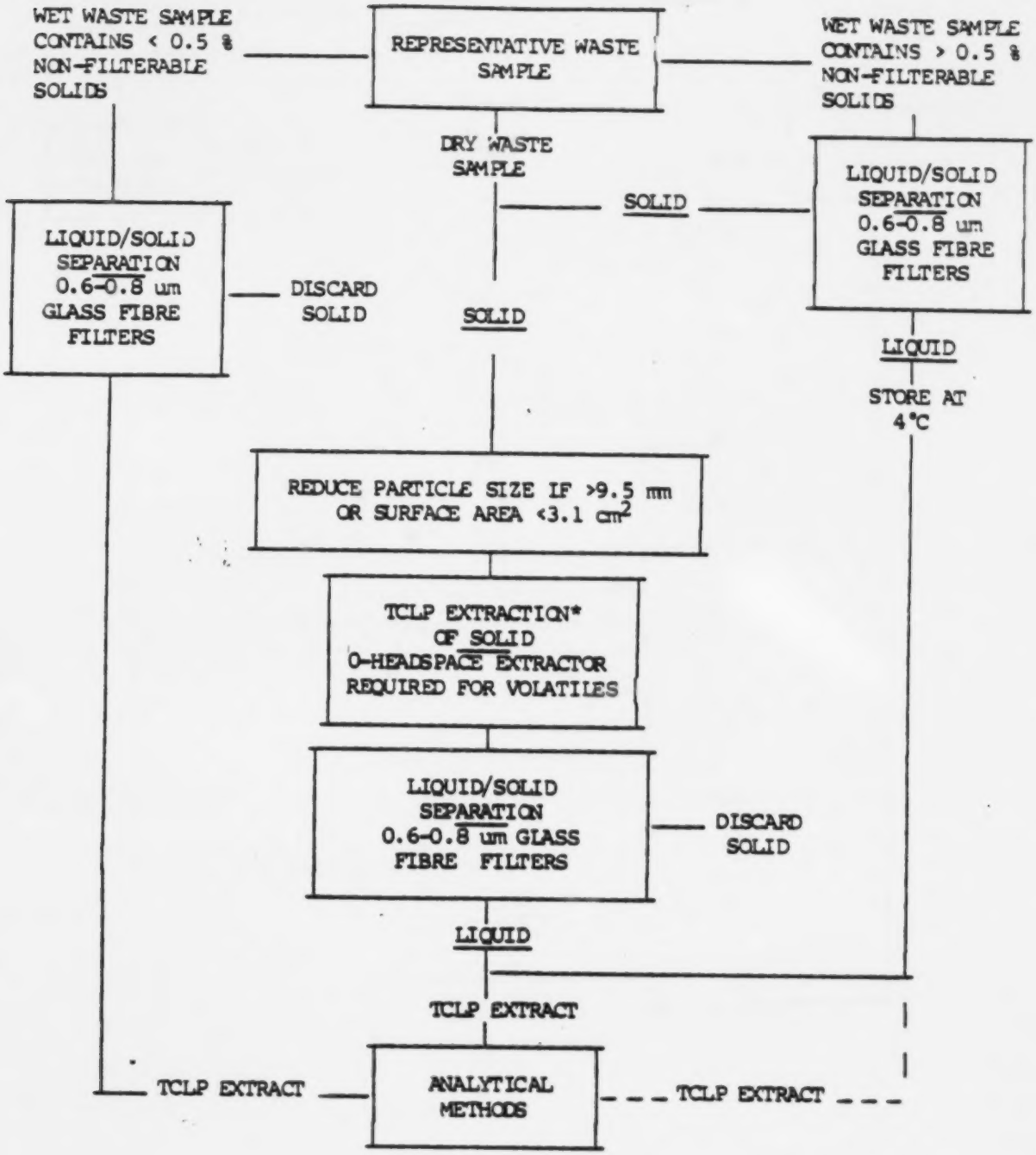


Figure 1: Extraction Procedure Flowchart.

FIGURE 2 : TCLP Flowchart



* The extraction fluid employed is a function of the alkalinity of the solid phase of the waste.

not migrate in total).

In addition, since different analysts may expend varying degrees of effort in accomplishing the liquid/solid separation with these waste types, this problem also contributes to variability. As indicated below, EPA believes that the liquid/solid separation technique that has been developed for the TCLP protocol reduces the variability that was associated with the EP's liquid/solid separation technique, and that it also provides a more adequate differentiation between those materials that behave as liquids in the environment, and those materials which behave as solids.

Initially, it was felt that this problem could be addressed through use of the much simplier liquid/solid separation technique used in RCRA Test Method 9095 (Paint Filter Free Liquid Test) (U.S. EPA, 1982). This method involves gravity filtration through a 60 mesh paint filter. This test method was promulgated on April 30, 1985 (50 FR 18370). It is intended to be a qualitative determination of whether a waste contains any free liquids, and was developed in response to bans instituted on the disposal of liquids in landfills.

In applying this method to the TCLP, however, a number of problems were encountered (ERCO, 1984). The most serious of these was the fact that particulates, which are solids, are capable of passing through the paint filter in bulk. Using Method 9095 in the TCLP, would lead to these solids being considered as a liquid, and thus, not subject to extraction. This could lead to an artificially high (or low) apparent extract concentration. In addition, the amount of liquid the method

yields varies with how the waste is poured or placed in the filter. These two problems negated the use of Method 9095 in the TCLP.

To overcome the problems encountered with the paint filter method, EPA has returned to the use of pressure filtration to separate the liquid from the solid phase of a waste. However, in re-evaluating this technique, several changes have been made which will decrease the time it takes to accomplish separation, improve the precision of the method, and provide a more adequate differentiation between those materials which behave as liquids in the environment and those which behave as solids. These changes include switching from a 0.45 um filter medium of varying composition, to specifying a 0.6-0.8 um glass fiber filter (nominal pore size), as well as limiting the time spent filtering. The use of glass fiber will also reduce the possibility of adsorption of analytes to the filter media. Also, these filters have a much higher throughput and show much less tendency to clog, and for these reasons, allow the use of a pressure of 50 psi rather than 75 psi to accomplish separation. Initial experiments indicate substantial operational advantages and time savings with the use of glass fiber filters (ERCO, 1985), which should also help to improve precision. In addition, EPA believes that these filters will provide a more adequate differentiation between those materials which behave as liquids in the environment, and those which behave as solids.

C. Use of Extraction Devices

The EP protocol contains a descriptive definition of what

was considered to be acceptable agitation. Two types or extraction equipment are described in the EP protocol which were determined to meet this definition. One is a stirrer type extractor which uses small fan-like blades to mix the extraction fluid with the waste. The other type involves rotary action in which closed bottles containing the waste/extraction fluid mixture are tumbled in an end-over-end fashion (U.S. EPA, 1982). This lack of specificity in agitation conditions is also a major source of variability (Brown et al., 1983).

The TCLP protocol eliminates this source of variability by specifying a single means of agitation (i.e., rotary or tumbler), and a fixed agitation rate (30 $\pm$ 2 rpm). The rotary or tumbler type of extractor was selected for several reasons. It is widely recognized as a reproducible means of contacting the liquid and solid, and has been standardized by ASTM in their draft method D3987 (ASTM, 1985). Also a factor in this determination was that the Agency's Science Advisory Board, in reviewing the TCLP development program, recommended that EPA develop one device and set of operating conditions (SAB, 1984). Although EPA recognized that this would require some laboratories to purchase additional equipment, EPA has opted to propose the use of rotary agitation only.

Another aspect related to extraction devices deals with the need to prevent loss of volatile organic compounds during the conduct of the procedure. This includes losses during initial and final liquid/solid separation, extraction, and sample handling. With the assistance of laboratory equipment manufactures, EPA

has addressed this problem through development of a Zero-Headspace Extractor (ZHE). After experimentation with several prototype devices, the device described schematically in Figure 3 has been sucessfully applied during evaluation of the TCLP procedure (See Section VI of this background document). Equipment of this type is now available from two suppliers (See TCLP protocol provided in Section VII of this background document).

The ZHE is capable of conducting the intial liquid/solid separation, agitation, as well as final extract filtration, with only minimal loss of volatiles. Although considerably more expensive than the bottles used in the current EP, these devices are only required when investigating the leachability of volatile components. Less expensive vessels are used for assessing the mobility of non-volatile components. In addition, since the ZHE

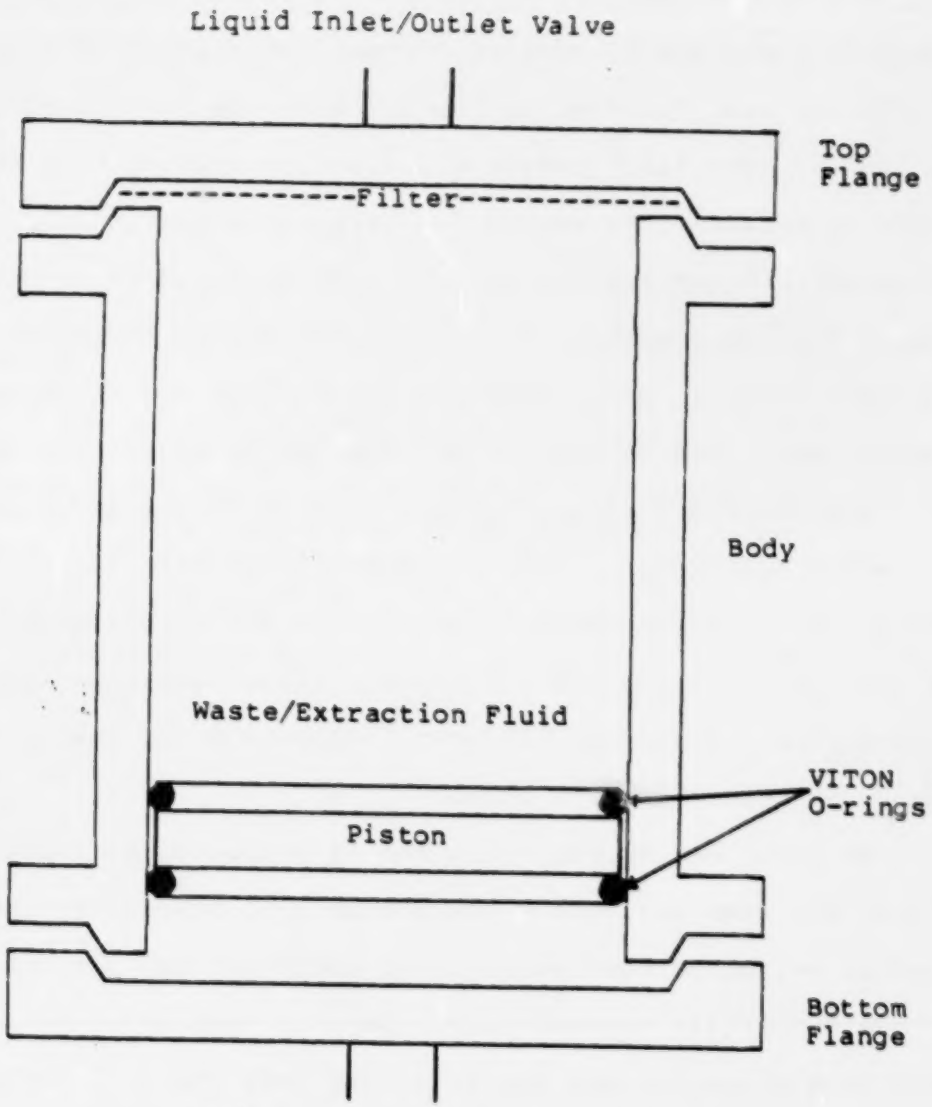


Figure 3: Zero-Headspace Extraction Vessel

is capable of also conducting the liquid/solid separation, no outside filtration apparatus is required.

Due to the need to have the ZHE compatible with common laboratory equipment, such as off-gassing ovens, and laboratory sinks, and also the need to produce a device that is easily handled by laboratory personnel, a device smaller than the 2 liter internal volume device EPA originally had in mind was necessary. Balancing the need to also accommodate as large a sample size as possible, EPA determined that a device with a one half liter internal internal volume (500 ml) would be most appropriate. Due to the 500 ml internal capacity, the ZHE can only accommodate a maximum sample size of 25 grams for a 100 percent solids sample. For a waste of less than 100 percent solids, the maximum sample size the ZHE can accommodate is tied to the percent solids of the waste. The device can only accommodate the minimal 100 gram sample size for wastes that are 25 percent solids or less.

As indicated above, while EPA is proposing to require use of the ZHE when evaluating the mobility of volatiles, the use of regular extractor bottles is being specified for use when dealing with non-volatile contaminants. Regular extraction bottles are much less expensive and easier to use than the ZHE. While EPA had originally intended the ZHE to be allowed to be used for non-volatile components as well, certain features of the device, and other constraints have led EPA to only allow its use when dealing with volatiles.

The problem touches upon the SAB's concern that, in the

interest of precision, one device and one set of operating conditions should be specified (SAB, 1984). There are actually two factors which differ between regular extraction bottles and the zero-headspace vessel which could affect precision. The first is that since regular extraction bottles will provide for at least some headspace, agitation is likely to be slightly greater than with the ZHE vessel.

The second factor is that the two devices involve slightly different types of liquid/solid separation. Whereas the ZHE requires piston-applied pressure, use of bottles involves conventional air pressure filtration. These two means of applying pressure to accomplish liquid/solid separation are capable of producing different results from some waste types, particularly for wastes that contain large (i.e., 9.5 mm) sized particles. (The TCLP protocol limits particle size to a maximum of 9.5 mm - See Section IV discussion regarding particle size reduction).

The problem with wastes that contain 9.5 mm particles and the use of piston pressure, is that liquid/solid separation could be prematurely halted when the piston contacts the largest waste particle. In this case, some portion of the intitial liquid phase of the waste will remain within the ZHE vessel. Although specifying a still smaller particle size (e.g., $\leq$ 1 mm) would eliminate this problem, this is not a good alternative since this could make the test unrealistically aggressive for some wastes.

Due to these factors, use of the ZHE is being limited to evaluation of volatile contaminants.

D. Other improvements

In addition to the major improvements discussed above, EPA has instituted a number of minor improvements into the TCLP protocol. These improvements are primarily designed to increase the overall precision of the method and to reduce the cost of the test. First, in transferring samples from container to filtration apparatus to extractor, etc., the TCLP procedure calls for determining the weight of any residual sample material left behind and subtracting this from the total sample size. This will insure that the amount of extracting medium added to the extractor is truly a function of the solid material within the extractor, and will help to improve overall precision.

Whereas agitation in the EP procedure was conducted over a period of 24 hours, the TCLP protocol is limited to 18 hours. Data obtained prior to the initiation of the TCLP development program indicated that equilibrium for most waste components was achieved within 18 hours of agitiation, and that when equilibrium was not achieved during this time period, it was likely that equilibrium would also not be achieved during longer agitiaion periods (Brown et. al., 1983). This change should help to reduce the overall cost of the method.

The TCLP procedure also contains a number of helpful hints designed to guide the analyst during certain steps that may be difficult or unclear. For example, while the use of glass fiber filters and the application of limits on the time spent filtering has helped to solve some of the operational difficulties of the TCLP protocol, the analyst is cautioned to not subject these

filters to instantaneous high pressures. While some analysts may be prone to do this in an effort to save time, they may not be aware that the instantaneous application of high pressures may degrade the filters to the point that they may no longer be able to adequately separate the liquid from the solid phase of the waste.

IV. OTHER ASPECTS

A. Particle Size Reduction

The EP protocol requires particle size reduction in those cases where the waste cannot pass through a 9.5 mm sieve, or has a surface area of greater than 3.1 cm^2/gm. The TCLP continues with this requirement. One difference, however, deals with particle size reduction for monolithic wastes. The EP allows the alternative of using the Structural Integrity Procedure (SIP), which amounts to pounding the monolithic waste with hammer-like blows and then conducting the extraction on the resulting sample, whether in one piece or in many pieces. The proposed TCLP does not allow use of the SIP (i.e., requires particle size reduction) for several reasons. The first reason again has to do with precision and the Science Advisory Board's comment to limit the new procedure to one device and set of operating conditions.

Secondly, the Agency believes that given the uncertainties concerning the long term environmental stability of solidified wastes, an environmentally conservative approach is warranted. The SIP was originally developed as a means of assessing the degree to which a cementitious process stabilized a waste to the extent that the waste would remain as a monolithic block even after disposal. Such stabilization processes decrease

leaching potential through reduction of surface area, and thus the area of potential leachate contact. Many processes also provide for chemical stabilization by binding heavy metals in insoluble hydroxide and other complexes.

The Agency believes that physical stabilization alone is not enough to insure that components do not leach in significant quantities from wastes. There are two types of actions which may act to reduce the physical integrity of stabilized wastes. First, the action of heavy landfill equipment, which the SIP is designed to simulate, will act to reduce the monolithic blocks into smaller pieces. Secondly, and more important, is the affect of natural weathering forces, such as wet/dry and freeze/thaw cycles (Hannack, 1985). The SIP does not account for such weathering. The Agency is currently investigating the effects of natural weathering on monolithic wastes, and may propose the use of additional predictive methodology at some later date. In the interim, by not allowing the use of the SIP, the Agency insures that generators do not rely on physical stabilization alone as a means of waste treatment.

An unrelated problem regarding particle size reduction also involves the treatment of volatile contaminants. While EPA is attempting to prevent loss of volatiles (through introduction of the ZHE), if a waste containing volatiles requires particle size reduction, it is likely that some portion of these volatiles will be lost prior to their introduction into the ZHE.

Herein lies a problem that may require a trade-off. Is it more important to reduce particle size or to prevent loss of

volatiles? EPA believes that particle size reduction is more important and has addressed this problem in the proposed TCLP by specifying that, where possible, particle size reduction be conducted to the extent possible on the sample as it is being taken.

The protocol does recognize, however, that there will be situations where volatile containing samples requiring particle size reduction cannot be reduced under these conditions. In this case, the protocol specifies that where particle size reduction must be done in the laboratory, the sample should first be refrigerated to reduce the vapor pressure of the volatiles, and that any reduction equipment used be refrigerated as well. (The TCLP also limits reduction equipment to those which do not generate heat). In addition, the protocol cautions that particle size be reduced with minimal exposure of the waste to the atmosphere. These actions should help to minimize loss of volatiles if reduction must be conducted in the laboratory.

B. Treatment of Alkaline Wastes

As mentioned previously, alkaline wastes were not adequately represented by the eleven wastes used in the TCLP development program. EPA is concerned that the potential hazard posed by these wastes may be underestimated by the acetate buffer system initially chosen for the TCLP (Kimmell and Friedman, 1984). Specifically, EPA believes that an increase in the leaching of inorganic and some organic species may be observed as the ability of the waste to resist the acidity encountered during leaching becomes exausted. Note that this can occur well after the 20 to

1 liquid to solid ratio (leaching interval) selected for both the EP and the TCLP. Data from the TCLP development program (on a moderately alkaline waste), and from subsequent studies on wastes of moderate to high alkalinity, demonstrated that the leaching rate of metals was relatively constant or increasing slightly over liquid to solid ratios as high as 30 to 1 (Francis et. al., 1986(a)). This data demonstrated that the TCLP acetate buffer may not adequately account for the leaching of some metals from waste of moderate to high alkalinity.

To address this problem, EPA determined that an increase in the acidity of the leaching media for the alkaline wastes would adequately account for the increased leaching of these species that could eventually occur in landfills. To define this second leaching fluid, the basis behind the EP's maximum amount of acetic acid (i.e., two milliequivalents of acid per gram of waste), was used. Data gathered at EPA's Boone County Field Site over a period of seven years indicated that the leachate generated by decomposing municipal waste contains approximately 0.14 equivalents of acidity per kilogram of dry refuse. Applying this data to the hypothetical codisposal environment, EPA concluded that 1 gram of industrial waste could potentially be acted upon by 2 milliequivalents of acid. For a one hundred gram sample (the EP's minimum sample size), this translated to a total of 200 milliequivalents of acid (U.S. EPA, 1980). The acetate buffer system originally chosen for the TCLP supplies only 70 milliequivalents of acid for a 100 gram sample.

As indicated above, steady or increased leaching of inorganic species was demonstrated to occur up to and after the 20 to 1 liquid to solid ratio (Francis et al., 1986(a)). While this data demonstrates that the 70 milliequivalent acetate buffer system is not aggressive enough for most of the inorganic species investigated, it supports the use of a 200 milliequivalent acetic acid solution for only some of the inorganic species. The Agency is, however, proposing use of the 200 milliequivalent acetic acid solution for alkaline wastes to be protective of human health and the environment when such leaching does occur. The Agency believes this action is justified given the conservative nature of the Hazardous and Solid Waste Amendments of 1984. In addition, as indicated in Francis et al., 1984, municipal waste leachates, both those generated in lysimeters and real leachates, have been observed in other studies to contain higher concentrations of carboxylic acids (measured as total organic carbon, of which approximately 70 percent is made up of carboxylic acids (Francis et al., 1984)), than those measured in the municipal waste leachate used in the TCLP development program.

Hence, EPA is proposing a two leaching fluid system for the TCLP. As explained above, the Agency has chosen to base its choice of the strength of the alkaline waste leaching medium on the basis behind the EP's limit on the amount of acetic acid used. This will involve a 2 milliequivalent of acid per gram of waste leaching fluid for wastes of moderate to high alkalinity, and a 0.7 milliequivalent of acid per gram of waste leaching fluid for other wastes.

A simple test of waste alkalinity is proposed as a means of determining the appropriate leaching fluid. This test involves first mixing a small subsample of the solid phase of the waste with water at approximately a 20 to 1 liquid to solid ratio, and measuring the pH. If the pH is less than or equal to 5.0, the waste is primarily acidic in character (has little if any buffering capacity) and the TCLP acetate buffer leaching media is used. If the pH is greater than 5.0, 0.7 milliequivalents of acid per gram of subsample (that used in the TCLP acetate buffer leaching fluid), is added to the slurry, heated for a 10 minute period, cooled, and the pH is again measured. Again, if the pH is less than or equal to 5.0, the waste has little alkaline character (limited buffering capacity), and the TCLP acetate buffer leaching media is used. If the pH is above 5.0, this demonstrates that the waste, when exposed to 0.7 milliequivalents of acid per gram of subsample, has significant alkaline character and that the more acidic (i.e., 2.0 milliequivalent of acid per gram of waste) leaching fluid is used. The development of this simple test and the make-up of both of the TCLP leaching fluids is discussed more fully in ERCO, 1986.

Note that EPA is not proposing this dual leaching fluid system for the evaluation of volatile compounds. These compounds are not expected to be affected by slight changes in acidity, as demonstrated by data obtained during the TCLP development program (Francis et al., 1984, Francis et al., Feb., 1986, and TAI, May, 1985).

C. Use of a Pre-Screen Test

While the TCLP is expected to be similar to the EP in cost, since the TCLP will involve the analysis of a number of additional contaminants, including volatiles, the overall cost of evaluating wastes using the TCLP will be considerably more than that using the EP (i.e., $2000 - $4000 for the TCLP as opposed to about $1000 for the EP). To cut these analytical costs while at the same time not sacrifice anything in terms of environmental protection, EPA is proposing to establish an optional prescreen test for the TCLP protocol. This prescreen test will consist of a total analysis of the waste, using SW-846 methods (U.S. EPA, 1982), to determine if the waste contains sufficient amounts of individual contaminants for the appropriate regulatory threshold to be exceeded, assuming that all the contaminant leaches from the waste. If based on such an analysis, one can be certain that the appropriate regulatory threshold cannot be exceeded, then the TCLP does not have to be performed.

For example, let us assume that a generator wishes to show that his waste contains insufficient amounts of compounds A and B for the regulatory threshold to be exceeded. Also assume that the regulatory threshold for both compounds is 10 mg/l, and that the waste is a solid material (i.e., no liquid phase). The generator takes representative subsamples of the waste in question, performs the appropriate SW-846 analyses, and concludes that the waste contains an upper limit of approximately 22 mg of compound A and 8 mg of compound B in a 100 gram sample. Assuming that all of both compounds leach from the waste, and given that the

100 gram sample of waste would be leached with 2 liters of extraction fluid (from the 20 to 1 liquid to solid ratio), this translates to a maximum TCLP extract concentration of 11 mg/l of Compound A and 4 mg/l of compound B. Given this analysis, the generator would conclude that while the waste could not exceed the threshold for compound B, it could exceed the threshold for Compound A. In this case, it might be prudent for the generator to conduct the TCLP for Compound A, as it may turn out that not all the compound is actually leachable, as determined by the TCLP.

Note that in the above example, if the waste in question were all liquid (i.e., no solid phase), since the TCLP (as well as the EP) treates liquid wastes as their own extract, the waste would already exceed the threshold for compound A.

D. Quality Assurance Requirements

The quality assurance (QA) requirements of the EP are relatively straight-forward. The require a minimum of one blank per sample batch, and the method of standard addition (MSA) to be run for all samples. The Agency has received comments that the requirements with respect to the use of blanks is unclear and that requiring MSA for all extractions, which is very expensive, is unnecessary for all situations. This is particularly significant in determining the quality assurance requirements for the TCLP, given the increased number of analytes. In addition, the EP protocol is felt to need clarification and expansion in addressing other aspects of quality assurance, such as acceptable sample holding times, and use of replicates.

The QA requirements of the analytical methods used in analyzing the TCLP extracts are defined in the appropriate SW-846 methods.

The use of blanks (or controls) is well recognized in experimental design as a means of determining whether observed test results are different form those that would be expected in the absence of some test condition. Testing under the TCLP is no different. Blanks are necessary to determine whether the results observed are true results, or whether contamination of either laboratory extraction equipment or the extracting fluid might be contributing to the observed results. Hence, the QA requirements specify that extraction equipment should be periodically (i.e., one blank for every 10 extractions) subjected to blanks as a check for any contamination (memory effects from previous testing), and that every batch of leaching fluid made up should also be subject to a blank.

The method of standard additions is used to determine if interferences are present in the extract, such that the analytical method may be producing inaccurate results. MSA, however, is expensive. Therefore, EPA is only requiring its use under two conditions: 1) When recovery of a particular compound from spiked splits of an extract is not between 50 and 150 percent, or 2) When the concentration of the contaminant measured in the extract is within 20 percent of the appropriate regulatory threshold. These changes recognize that MSA is only necessary when the measured concentration of a contaminant is close enough to the threshold, that matrix interferences could yield a wrong decision regarding the determination of hazard, or that severe

matrix interferences are preventing the analytical method from accurately determining the concentration of the analyte.

Specifying holding times for the EP metals was not critical, since these species generally experience no significant degradation within normal analysis periods. Since the TCLP involves quite a few more organic compounds, including volatiles, sample holding times begin to take on more importance. The sample holding times specified for the TCLP are consistent with those used for the Agency's Contract Laboratory Program.

Whereas the use of replicates was called for in earlier drafts of the TCLP (U.S. EPA, April, 1985 and U.S. EPA, October, 1985), the proposal draft requires no replicate testing. The purpose of requiring replicates is to insure that the waste in question has been adequately characterized. In other words, that representative samples of the waste have been taken and tested. While EPA believes that more than one sample is generally necessary to achieve adequate characterization, the determination of how many samples to test is a very waste specific exercise. Some wastes that are relatively homogeneous in nature may require only two or three samples to insure representativeness. Other wastes that are not homogeneous may require more. Given that the determination of how many samples constitute a representative determination is waste specific, the Agency feels that the determination of how many samples of a waste to test is best determined by those who are most knowledgable about the waste. Guidance concerning representative sampling is presented in the current edition of EPA's solid waste testing manual SW-846

(U.S. EPA, 1982). EPA is currently updating this section of the manual to provide more guidance, and a draft is available (U.S. EPA, December, 1985). This draft also provides guidance regarding how often to test.

V. PUBLIC PARTICIPATION

In an effort to identify and resolve any potential problems associated with the TCLP protocol prior to proposal, and also to inform the interested public of the Agency's activities in this area, EPA held a number of meetings at which draft procedures were circulated (U.S. EPA, April, 1985 and U.S. EPA, October, 1985), and various aspects of the procedure were reviewed. These included public discussions at meetings of the Association of Official Analytical Chemsits (AOAC), the American Society of Testing and Materials (ASTM), and EPA's Annual Solid Waste Testing Symposium. In addition, drafts of the method were sent out to all State solid waste management aencies, and to others who requested the draft. Finally, EPA's collaborative study, which involves in excess of 20 laboratories, has provided an active forum for identifying and resolving potential problems associated with the TCLP protocol.

As a result of these activites, EPA received a number of comments and suggestions regarding the TCLP protocol, both verbally and written. All of these were given full consideration, and have resulted in various improvements in the proposed test method.

VI. TCLP EVALUATION - RESULTS OF PHASE III

Phase III of the TCLP Development program involved an evaluation of ruggedness and precision as well as a multi-laboratory collaborative study. The experimental design and a

summary of the results of the precision evaluations are presented below. While the ruggedness evaluation for the metals and semi-volatiles has been completed (ERCO, Feb. 21, 1986), the work on the volatiles portion of the method is in progress. The results will be noticed for comment upon completion.

EPA's collaborative study is currently ongoing. In addition, the Electric Power Research Institute (EPRI) has conducted a limited collaborative evaluation of the draft TCLP protocol, primarily as it applies to inorganic constituents. The results of both of these studies will be noticed for comment when completed.

A. Precision Evaluation

As discussed earlier, the TCLP protocol requires the use of a Zero-Headspace Extractor (ZHE) when dealing with volatiles, and the use of common EP extraction equipment (i.e., bottles) when dealing with non-volatile components. In response, EPA has conducted a precision evaluation of the TCLP protocol using both devices. These evaluations were conducted by two laboratories, each laboratory conducting a number of replicate extractions on two wastes. These wastes were an API separator sludge/ electroplating waste admixture containing nonvolatile organics and a variety of inorganics, and an ammonia still lime sludge containing a variety of polycyclic aromatic hydrocarbons, and several inorganic compounds. These wastes were also spiked with several volatile compounds.

The results of the precision evaluation for non-volatile components indicate the TCLP to be of acceptable precision (S-Cubed, January, 1986). For the most part, the percent coefficient

of variations between replicate extractions for individual constituents was less than 30 percent. This includes the variability contributed by sampling variability and analytical variability. Although sampling variability was minimized to the extent possible, it is reasonable to expect a sample variability contribution to the total variability of between 2 and 5 percent. Analytical variability, as demonstrated in the S-Cubed report, was in many cases comparable to, and in some cases exceeded, the total variability. This observation is significant as the analytical methods used to analyze the TCLP extracts are well accepted and in widespread use.

Precision for the non-volatiles was observed to be best for those contaminants present at relatively high levels, as is the usual case in any analysis for method precision. For those cases where the contaminant was present at relatively low concentrations, precision was fair, the precent coefficient of variation generally falling below 50 or 60 percent.

The results of the precision evaluations for the volatile components (Francis et al., 1986(b)), are not as clearly interpreted. There are several reasons for this. These evaluations were initiated as the zero-headspace extractor became available. Recall that the present design for the ZHE was the result of experimentation with several prototype devices. Hence, experience with the ZHE, especially by laboratory technicians who were responsible for conducting the work, was limited.

In addition, the precision work on the volatiles was conducted

using two draft TCLP protocols. The first public draft protocol was released for comment in April, 1985 (U.S. EPA, April, 1985). At this time EPA was still experimenting with several prototype devices, and although the April TCLP draft addressed volatile components, it was largely to obtain technical comments and suggestions, and was not based on an actual working ZHE device. It was this protocol under which the TCLP precision evaluation of the volatiles was begun.

The second public draft of the TCLP protocol was released for comment in October of 1985 (U.S. EPA, October, 1985). Although this draft was based on the current design for the ZHE, further experience with the device has led EPA to re-write the TCLP volatiles procedure in the form that it currently appears (51 FR 1750).

The remainder of the precision evaluation for the volatiles was conducted using the October draft TCLP. Several significant changes have been made in the current (proposed) version due to experience gained with the ZHE device. For example, whereas the October version allowed the use of VOA vials for the collection of the TCLP extract, the proposed method requires the use of air-tight syringes or TEDLAR® bags due to expected losses of volatiles from the VOA vials during collection of the extract. VOA vials were used to collect the TCLP extract during this evaluation.

AIso, in following the protocols, inadvertant errors were apparently made which seem to have affected method precision. For example, whereas the October version of the TCLP protocol

placed a maximum of 25 grams on the amount of solid material the ZHE could accommodate, considerably more solid material was extracted during the precision analysis of one of the wastes (i.e., the API separator sludge/electroplating waste admixture). This provided for a variable liquid to solid ratio rather than the specified 20 to 1 ratio.

To complicate matters further, due to extenuating circumstances, two individual laboratories conducted the analytical work rather than the intended single laboratory. It is apparant that higher concentrations were obtained on the same waste from the different laboratories.

As indicated above, these factors make the precision data difficult to interpret. Whereas the precent coefficient of variations on the ammonia still lime sludge were mostly less than 60 or 70 percent, which is fair given the nature of volatiles, the numbers generated from the admixture of API separator sludge and electroplating waste indicated more variability. As indicated in the report (Francis et al., 1986(b)), some of this can be attributed to the oily character of the waste which seemed to have dominated the extraction.

Due to the inconclusive nature of the results, EPA is in the process of conducting another precision evaluation of the volatile components. This study will use the proposal draft of the TCLP, which we believe should help to clear up some of the problems encountered during the first evaluation. This study will be similar to the previous one in most other aspects, except that a third waste will be evaluated (one expected to not react with

the spiked volatiles), and two levels of volatile spike will be used (i.e., one of relatively high concentration and one of relatively low concentration). The results of this evaluation will be made available to the public on its completion.

B. Ruggedness Evaluation

A ruggedness evaluation is designed to determine how sensitive a test method is with respect to modest departures from the protocol which can be expected during routine applications of the protocol. The purpose of this evaluation is to identify procedural variables which must be carefully controlled, and then to emphasize in the protocol the limits of acceptable deviation with respect to these variables. If a procedure is "rugged" it will be unaffected by minor departures from the specified method values. If results are affected by variation of conditions, the protocol must be written to specify those parameters which must not be varied beyond a determined amount.

As with the precision evaluation, ruggedness was evaluated for both the ZHE and common EP extractor bottles. Different lots of the same wastes used for the precision evaluations, were used for the ruggedness evaluation. These evaluations were performed by one laboratory. The ruggedness evaluation for the common (EP) extractor bottles has been completed (ERCO, Feb. 21, 1986).

Table 3 presents the parameters which were evaluated for ruggedness using both types of extraction equipment. There were several parameters which EPA intended to investigate (i.e., extraction temperature and agitation rate) which could not be accommodated due to lack of appropriate laboratory equipment necessary to vary these parameters. In addition, while EPA had

Table 3

Parameters Investigated During TCLP Ruggedness Evaluation

Parameter	TCLP Specification	ZHE Device	Common Equipment (Bottles)
1) Liquid/Solid ratio:	20	19 - 21	19 - 21
2) Extraction time:	18	--	16 - 20
3) Headspace: ZHE:	zero	0 - 5%	--
Bottles:	variable	--	20 - 60%
4) Medium #1 acidity: (milliequivalents acid)	70	60 - 80	--
5) Medium #2 acidity: (milliequivalents acid)	200	--	190 - 210
6) Aliquots: (taking of aliquots directly form ZHE for analysis)	Allowed for ZHE in some cases (See TCLP protocol in Section VII)	Yes - No	--
7) Extractor vessel:	(See TCLP protocol in Section VII)	Associated ZHE - Millipore ZHE	Borosilicate - Flint glass
8) Acid wash filters:	Required for metals	--	Yes - No
9) Filter Type:	0.6-0.8 um glass fiber	--	Glass fiber - Polycarbonate
10) Pressurization of ZHE during agitation (psi)	5 - 10	0 - 20	--
11) ZHE extract collection devices.	Tedlar bag or Syringe	Tedlar bag - Syringe	--

originally intended to evaluate the effect of different glass fiber filters (See Table 3, Item 9), glass fiber filters other than the filter type specified in the TCLP protocol were unable to withstand the pressures stipulated in the TCLP. Hence the EP's use of polycarbonate filters were investigated instead. EPA has already determined that extract concentrations may differ slightly between the two filter types (ERCO, 1985(a) and Francis et al., Feb., 1986). The remainder of the Table 3 parameters are largely self-explanatory.

The ruggedness evaluation for the common (EP) extraction equipment demonstrated that, for the most part, the TCLP is fairly rugged (ERCO, Feb. 21, 1986). This is especially true for the semi-volatile organics, which, with few exceptions, were unaffected by the parameters investigated. For the metals, the results suggest that at least two parameters may be critical. As expected, the acidity of the extraction fluid directly influences the extraction of metals. The TCLP protocol emphasizes accuracy in the preparation of the extraction fluids by specifying the exact recipes for the preparation of these fluids, and indicating that the pH of these fluids should be accurate to within $\pm$ 0.05 pH units.

Bottle type (i.e., borosilicate vs flint glass), is the second parameter which apparently affects the concentration of metals in the extract, and may also affect (to a lesser degree), the extraction of semi-volatiles. It appears that using flint glass can result in significantly higher extract concentrations. While acid washing the flint glass bottles, or an expanded use of blanks, may help to solve the problem, specifying borosilicate over other types of glass would

solve the problem entirely. Due to the substantially higher cost of the borosilicate glass (from 3 to 5 times higher), EPA is requesting comment on this option.

The volatiles evaluation for the TCLP is currently ongoing. As noted above, the Table D-3 parameters were investigated to determine if they need to be controlled more carefully. As an example, pressurization of the ZHE during agitation (Item 10), is being investigated to determine whether the build-up of pressure within the ZHE during agitation (which is expected to occur for some wastes, particularly carbonate containing waste), needs to be more carefully controlled. The build-up of this pressure could cause the ZHE piston to move, thereby causing the presence of headspace. The ruggedness evaluation would indicate if this variable should be controlled more carefully, perhaps by putting more pressure (e.g., 20 psi) behind the piston during agitation.

As indicated above, the results of the ruggedness evaluation for the volatiles will be noticed for comment upon completion.

C. Collaborative Study

As indicated earlier, both EPA and the Electric Power Research Institute (EPRI) have planned collaborative evaluations of the TCLP protocol. EPA's evaluation, in which a number of business associations and individual companies are participating, is currently ongoing. This study involves over 20 laboratories, five different wastes, both types of extraction equipment, and organic and inorganic compounds, including volatiles. The draft experimental design is discussed in U.S. EPA, February, 1986.

EPRI's study, which is very similar to an evaluation EPRI conducted on the EP (EPRI, 1979), was limited to the determination of inorganic compounds and deals with common extraction equipment only. This study deals with seven types of utility wastes and involves three laboratories. In addition to total precision, EPRI investigated the contribution of both variability in sampling, and variability introduced through the analytical methods, as was done during the investigation of the EP protocol. The draft experimental design for this collaborative evaluation is discussed in EPRI, July 10, 1985.

Both studies will be noticed for comment when completed.

VII. PROPOSED TCLP

2. Final Draft
2/20/85

APPENDIX II

Toxicity Characteristic Leaching Procedure (TCLP)

1.0 Scope and Application

1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present, but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

2.0 Summary of Method (See Figure 1)

2.1 For wastes containing less than 0.5% solids, the waste, after filtration through a 0.6-0.8 um glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than 0.5% solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced (if necessary), weighed, and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatiles (See Table 1). Following extraction, the liquid extract is separated from the solid phase by 0.6-0.8 um glass fiber filter filtration.

2.3 If compatible (e.g., precipitate or multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume weighted average concentration.

3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 Apparatus and Materials

4.1 Agitation Apparatus: An acceptable agitation apparatus is one which is capable of rotating the extraction vessel in an end over end fashion (See Figure 2) at 30 $\pm$ 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessel:

4.2.1 Zero-Headspace Extraction Vessel (ZHE). When the waste is being tested for mobility of any volatile contaminants (See Table 1), an extraction vessel which allows for liquid/solid separation within the device, and which effectively precludes headspace (as depicted in Figure 3), is used. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without having to open the vessel (See Section 4.3.1). These vessels shall have an internal volume of 500 to 600 ml and be equipped to accommodate a 90 mm filter. Suitable ZHE devices known to EPA are identified in Table 3. These devices contain viton O-rings which should be replaced frequently.

4.2.2 When the waste is being evaluated for other than volatile contaminants, an extraction vessel which does not preclude headspace (e.g., 2-liter bottle) is used. Suitable extraction vessels include bottles made from various materials, depending on the contaminants to be analyzed and the nature of the waste (See Section 4.3.3). These bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices:

4.3.1 Zero-Headspace Extractor Vessel (See Figure 3): When the waste is being evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter, and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the extract.

4.3.2 Filter Holder. When the waste is being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures up to 50 psi and more. The type of filter holder used depends on the properties of the material to be filtered (See Section 4.3.3). These devices shall have a minimum internal volume of 300 ml and be equipped to accommodate a minimum filter size of 47 mm. Filter holders known to EPA to be suitable for use are shown in Table 4.

4.3.3 Materials of Construction. Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used when evaluating the mobility of metals.

4.4 Filters: Filters shall be made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6-0.8 um, or equivalent. Filters known to EPA to meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid washed prior to use by rinsing with 1.0 N nitric acid followed by three consecutive rinses with deionized distilled water (minimum of 500 ml per rinse). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: Any of the commonly available pH meters are acceptable.

4.6 ZHE extract collection devices: TEDLAR® bags or glass, stainless steel or PTFE gas tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.

4.7 ZHE extraction fluid collection devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a constant displacement pump, a gas tight syringe, pressure filtration unit (See Section 4.3.2), or another ZHE device).

4.8 Laboratory balance: Any laboratory balance accurate to within $\pm$ 0.01 grams may be used (all weight measurements are to be within $\pm$ 0.1 grams).

5.0 Reagents

5.1 Water: ASTM Type 1 deionized, carbon treated, decarbonized, filtered water (or equivalent water that is treated to remove volatile components) shall be used when evaluating wastes for volatile contaminants. Otherwise, ASTM Type 2 deionized distilled water (or equivalent) is used. These waters should be monitored periodically for impurities.

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5.2 1.0 N Hydrochloric acid (HCl) made from ACS Reagent grade.

5.3 1.0 N Nitric acid (HNO_3) made from ACS Reagent grade.

5.4 1.0 N Sodium hydroxide (NaOH) made from ACS Reagent grade.

5.5 Glacial acetic acid (HOAc) made from ACS Reagent grade.

5.6 Extraction fluid:

5.6.1 Extraction fluid #1: This fluid is made by adding 5.7 ml glacial HOAc to 500 ml of the appropriate water (See Section 5.1), adding 64.3 ml of 1.0 N NaOH, and diluting to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

5.6.2 Extraction fluid #2: This fluid is made by diluting 5.7 ml glacial HOAc with ASTM Type 2 water (See Section 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 $\pm$ 0.05.

NOTE: These extraction fluids shall be made up fresh daily. The pH should be checked prior to use to insure that they are made up accurately, and these fluids should be monitored frequently for impurities.

5.7 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples shall be collected using a sampling plan that addresses the considerations discussed in "Test Methods for Evaluating Solid Wastes" (SW-846).

6.2 Preservatives shall not be added to samples.

6.3 Samples can be refrigerated unless it results in irreversible physical changes to the waste.

6.4 When the waste is to be evaluated for volatile contaminants, care must be taken to insure that these are not lost. Samples shall be taken and stored in a manner which prevents the loss of volatile contaminants. If possible, any necessary particle size reduction should be conducted as the sample is being taken (See Step 8.5). Refer to SW-846 for additional sampling and storage requirements when volatiles are contaminants of concern.

6.5 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. If they

need to be stored, even for a short period of time, storage shall be at 4°C and samples for volatiles analysis shall not be allowed to come into contact with the atmosphere (i.e., no headspace).

7.0 Procedure When Volatiles Are Not Involved

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration), is extracted. This will insure that there is adequate extract for the required analyses (e.g., semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (See Step 7.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (100 gram minimum) and proceed to Step 7.11.

7.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device discussed in Section 4.3.2, and is outlined in Steps 7.3 to 7.9.

7.3 Pre-weigh the filter and the container which will receive the filtrate.

7.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (See Section 4.4).

7.5 Weigh out a representative subsample of the waste (100 gram minimum) and record weight.

7.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.7 Transfer the waste sample to the filter holder.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.5, to determine the weight of the waste sample which will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., does not result in any additional filtrate within any 2 minute period), filtration is stopped.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter, and may cause premature plugging.

7.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying vacuum or pressure filtration, as outlined in Step 7.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid.

7.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 7.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (See Step 7.15) or stored at 4°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.5 or 7.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is less than 75 grams, review Step 7.0.

7.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has greater than 0.5% solids go to Step 7.11. If it appears that the solid may comprise less than 0.5% of the total waste, the percent solids will be determined as follows:

7.10.1 Remove the solid phase and filter from the filtration apparatus.

7.10.2 Dry the filter and solid phase at 100 ±

20°C until two successive weighings yield the same value. Record final weight.

7.10.3 Calculate the percent solids as follows:

$$\frac{\text{weight of dry waste and filters} - \text{tared weight of filters}}{\text{initial weight of waste (Step 7.5 or 7.7)}} \times 100 = \% \text{ solids}$$

7.10.4 If the solid comprises less than 0.5% of the waste, the solid is discarded and the liquid phase is defined as the TCLP extract. Proceed to Step 7.14.

7.10.5 If the solid is greater than or equal to 0.5% of the waste, return to Step 7.1, and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

NOTE: This step is only used to determine whether the solid must be extracted, or whether it may be discarded unextracted. It is not used in calculating the amount of extraction fluid to use in extracting the waste, nor is the dried solid derived from this step subjected to extraction. A new sample will have to be prepared for extraction.

7.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm^2, or is capable of passing through a 9.5 mm (0.375 inch) standard sieve, proceed to Step 7.12. If the surface area is smaller or the particle size is larger than that described above, the solid material is prepared for extraction by crushing, cutting, or grinding the solid material to a surface area or particle size as described above. When surface area or particle size has been appropriately altered, proceed to Step 7.12.

7.12 This step describes the determination of the appropriate extracting fluid to use (See Sections 5.0 and 7.0).

7.12.1 Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 gram portion to a 500 ml beaker or erlenmeyer flask.

7.12.2 Add 96.5 ml distilled deionized water (ASTM Type 2), cover with watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is $\leq$ 5.0, extraction fluid #1 is used. Proceed to Step 7.13.

7.12.3 If the pH from Step 7.12.2 is > 5.0, add 3.5 ml 1.0 N HCl, slurry for 30 seconds, cover with a watchglass, heat to 50°C, and hold for 10 minutes.

7.12.4 Let the solution cool to room temperature and record pH. If pH is $\leq$ 5.0, use extraction fluid #1. If the pH is > 5.0, extraction fluid #2 is used.

7.13 Calculate the weight of the remaining solid material by subtracting the weight of the sub-sample taken for Step 7.12, from the original amount of solid material, as obtained from Step 7.1 or 7.9. Transfer remaining solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid (See Step 7.12), into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close extractor bottle tightly, secure in rotary extractor device and rotate at 30 $\pm$ 2 rpm for 18 hours. The temperature shall be maintained at 22 $\pm$ 3 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle (due to the evolution of gasses such as carbon dioxide). To relieve these pressures, the extractor bottle may be periodically opened and vented into a hood.

7.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Step 7.7. This new filter shall be acid washed (See Section 4.4) if evaluating the mobility of metals.

7.15 The TCLP extract is now prepared as follows:

7.15.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 7.14 is defined as the TCLP extract. Proceed to Step 7.16.

7.15.2 If compatible (e.g., will not form precipitate or multiple phases), the filtered liquid resulting from Step 7.14 is combined with the initial liquid phase of the waste as obtained in Step 7.9. This combined liquid is defined as the TCLP extract. Proceed to Step 7.16.

7.15.3 If the initial liquid phase of the waste, as obtained from Step 7.9, is not or may not be compatible with the filtered liquid resulting from Step 7.14, these liquids are not combined. These liquids are collectively defined as the TCLP extract, are analyzed separately, and the results are combined mathematically. Proceed to Step 7.16.

7.16 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods identified in Appendix III of 40 CFR 261. TCLP extracts to be analyzed for metals shall be acid digested. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses, and combine the results mathematically by using a simple weighted average:

$$\text{Final Contaminant Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (l)
C_1 = The concentration of the contaminant of concern in the first phase (mg/l)
V_2 = The volume of the second phase (l)
C_2 = The concentration of the contaminant of concern in the second phase (mg/l)

7.17 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

8.0 Procedure When Volatiles Are Involved

The ZHE device has approximately a 500 ml internal capacity. Although a minimum sample size of 100 grams was required in the Section 7 procedure, the ZHE can only accommodate a maximum 100 percent solids sample of 25 grams, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Step 8.4 provides the means by which to determine the approximate sample size for the ZHE device.

Although the following procedure allows for particle size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible, any necessary particle size reduction (See Step 8.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the

waste to be exposed to the atmosphere for any more time than is absolutely necessary.

8.1 Pre-weigh the (evacuated) container which will receive the filtrate (See Section 4.6), and set aside.

8.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston O-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

8.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (25 gram maximum - See Step 8.0), record weight, and proceed to Step 8.5.

8.4 This step provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multiphasic, follow the procedure outlined in Steps 7.2 to 7.9 (using the Section 7 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If the waste obviously contains greater than 0.5% solids, go to Step 8.4.2. If it appears that the solid may comprise less than 0.5% of the waste, go to Step 8.4.1.

8.4.1 Determine the percent solids by using the procedure outlined in Step 7.10. If the waste contains less than 0.5% solids, weigh out a new 100 gram minimum representative sample, proceed to Step 8.7, and follow until the liquid phase of the waste is filtered using the ZHE device (Step 8.8). This liquid filtrate is defined as the TCLP extract, and is analyzed directly. If the waste contains greater than or equal to 0.5% solids, repeat Step 8.4 using a new 100 gram mimumum sample, determine the percent solids, and proceed to Step 8.4.2.

8.4.2 If the sample is $\leq$ 25% solids, weigh out a new 100 gram minimum representative sample, and proceed to Step 8.5. If the sample is > 25% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Step 8.4. Weigh out a new representative sample of the determined size.

8.5 After a representative sample of the waste (sample size determined from Step 8.4) has been weighed out and recorded, the sample is now evaluated for particle size (See Step 8.0). If the solid material within the waste obviously has a surface area per gram of material equal to or greater than 3.1 cm^2,

or is capable of passing through a 9.5 mm (0.375 inch) standard sieve, proceed immediately to Step 8.6. If the surface area is smaller or the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving (or equivalent means), and the solid is prepared for extraction by crushing, cutting, or grinding to a surface area or particle size as described above.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. Grinding and milling machinery which generates heat shall not be used for particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

When surface area or particle size has been appropriately altered, the solid is recombined with the rest of the waste.

8.6 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration.

8.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturers instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 8.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to slowly force all headspace out of the ZHE device. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

8.8 Attach evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10

psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within any 2 minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained less than 0.5% solids, (See Step 8.4) this filtrate is defined as the TCLP extract, and is analyzed directly - proceed to Step 8.13.

8.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 8.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (See Steps 8.13 and 8.14), or stored at 4°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (See Step 8.4). Record the final weight of the liquid and solid phases.

8.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.6).

8.11.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

8.11.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve, and disconnect the extraction fluid line. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion 2 or 3 times. Re-position the ZHE in the vertical position with the liquid inlet/outlet valve on top. Put 5-10 psi behind the piston (if necessary), and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to insure that they are closed.

8.11.3 Place the ZHE in the rotary extractor apparatus (if it is not already there), and rotate the ZHE at 30 $\pm$ 2 rpm for 18 hours. The temperature shall be maintained at 22 $\pm$ 3°C during agitation.

8.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve, and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Replace ZHE O-rings or other fittings, as necessary, and redo the extraction with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag, gas-tight syringe) holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Step 8.8. All extract shall be filtered and collected if the extract is multi-phasic or if the waste contained an initial liquid phase.

NOTE: If the glass fiber filter is not intact following agitation, the filtration device discussed in the NOTE in Section 4.3.1 may be used to filter the material within the ZHE.

8.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 8.12, and the initial liquid phase (Step 8.8) are collectively defined as the TCLP extract.

8.14 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods, as identified in Appendix III of 40 CFR 261. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses and combine the results mathematically by using a simple volume weighted average:

$$\text{Final Contaminant Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (l)
C_1 = The concentration of the contaminant of concern in the first phase (mg/l)
V_2 = The volume of the second phase (l)
C_2 = The concentration of the contaminant of concern in the second phase (mg/l)

8.15 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

9.0 Quality Assurance Requirements

9.1 All data, including quality assurance data, should be maintained and available for reference or inspection.

9.2 A minimum of one blank for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment is occuring. One blank shall also be employed for every new batch of leaching fluid that is made up.

9.3 All quality control measures described in the appropriate analytical methods shall be followed.

9.4 The method of standard addition shall be employed for each waste type if: 1) Recovery of the compound from spiked splits of the TCLP extract is not between 50 and 150%, or 2) If the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than 1 extraction is being run on samples of the same waste, the method of standard addition need only be applied once and the percent recoveries applied to the remainder of the extractions.

9.5 TCLP extracts shall be analyzed within the following periods after generation: Volatiles - 14 days, Semi-volatiles - 40 days, Mercury - 28 days, and other Metals - 180 days.

Table 1: Volatile Contaminants

Compound	CASNO
Acetone	67-64-1
Acrylonitrile	107-13-1
Benzene	71-43-2
n-Butyl alcohol	71-36-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

Table 2: Suitable Rotary Agitation Apparatus[1]

Company	Location	Model
Associated Design and Manufacturing Company	Alexandria, Virginia (703)549-5999	4-vessel device
		6-vessel device
Lars Lande Manufacturing	Whitmore Lake, Michigan (313)449-4116	10-vessel device
IRA Machine Shop and Laboratory	Santurce, Puerto Rico (809)752-4004	16-vessel device
EPRI Extractor	----------	6-vessel device[2]

[1] Any device which rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

[2] Although this device is suitable, it is not commercially made. It may also require retrofitting to accommodate ZHE devices.

Table 3: Suitable Zero-Headspace Extractor Vessels

Company	Location	Model No.
Associated Design and Manufacturing Company	Alexandria, Virginia (703)549-5999	3740-ZHB
Millipore Corporation	Bedford, Massachusettes (800)225-3384	SD1P581C5

Table 4: Suitable Filter Holders[1]

Company	Location	Model	Size
Nuclepore Corporation	Pleasanton, California (800)882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, California (415)828-6010	302400	142 mm
Millipore Corporation	Bedford, Massachusettes (800)225-3384	YT30142HW XX1004700	142 mm 47 mm

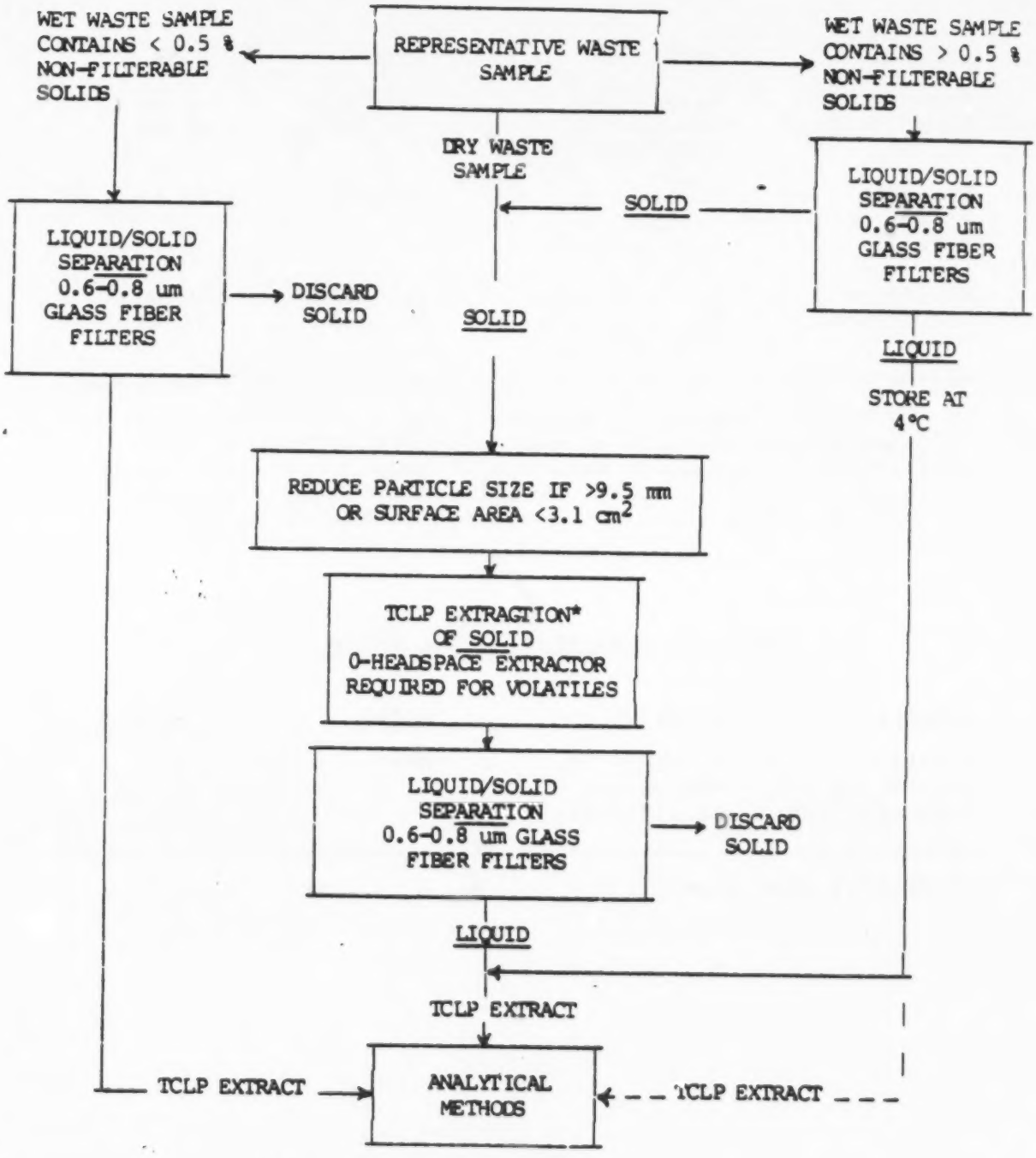
[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern.

Table 5: Suitable Filter Media

Company	Location	Model	Pore Size[1]
Whatman Laboratory Products, Inc.	Clifton, New Jersey (201)773-5800	GFF	0.7

[1] Nominal pore size.

FIGURE 1: TCLP Flowchart



* The extraction fluid employed is a function of the alkalinity of the solid phase of the waste.

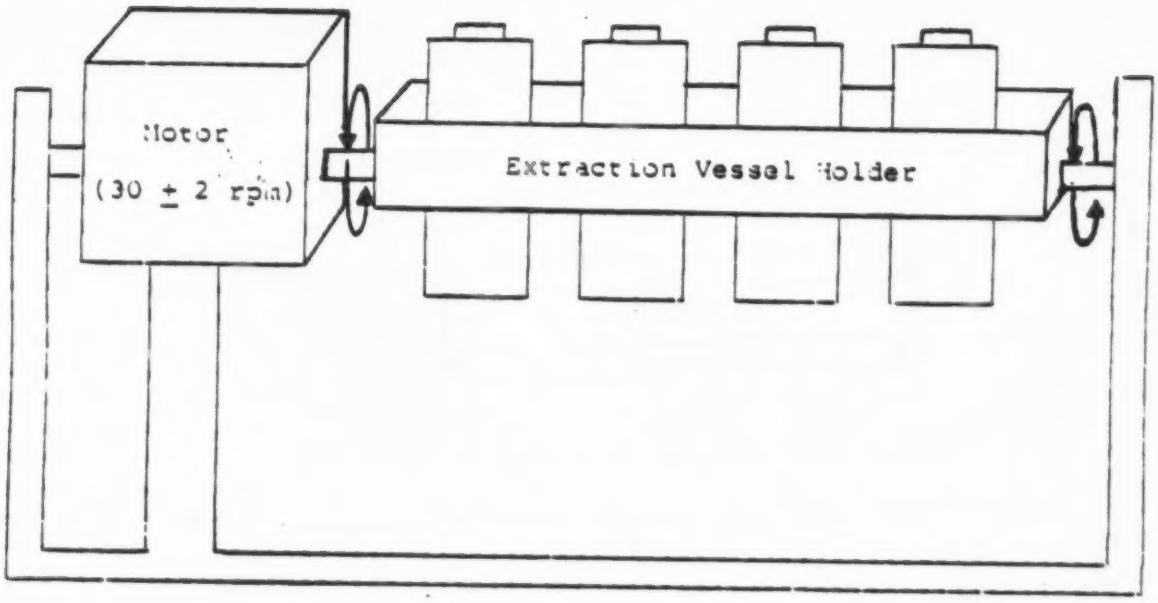


Figure 2: Rotary Agitation

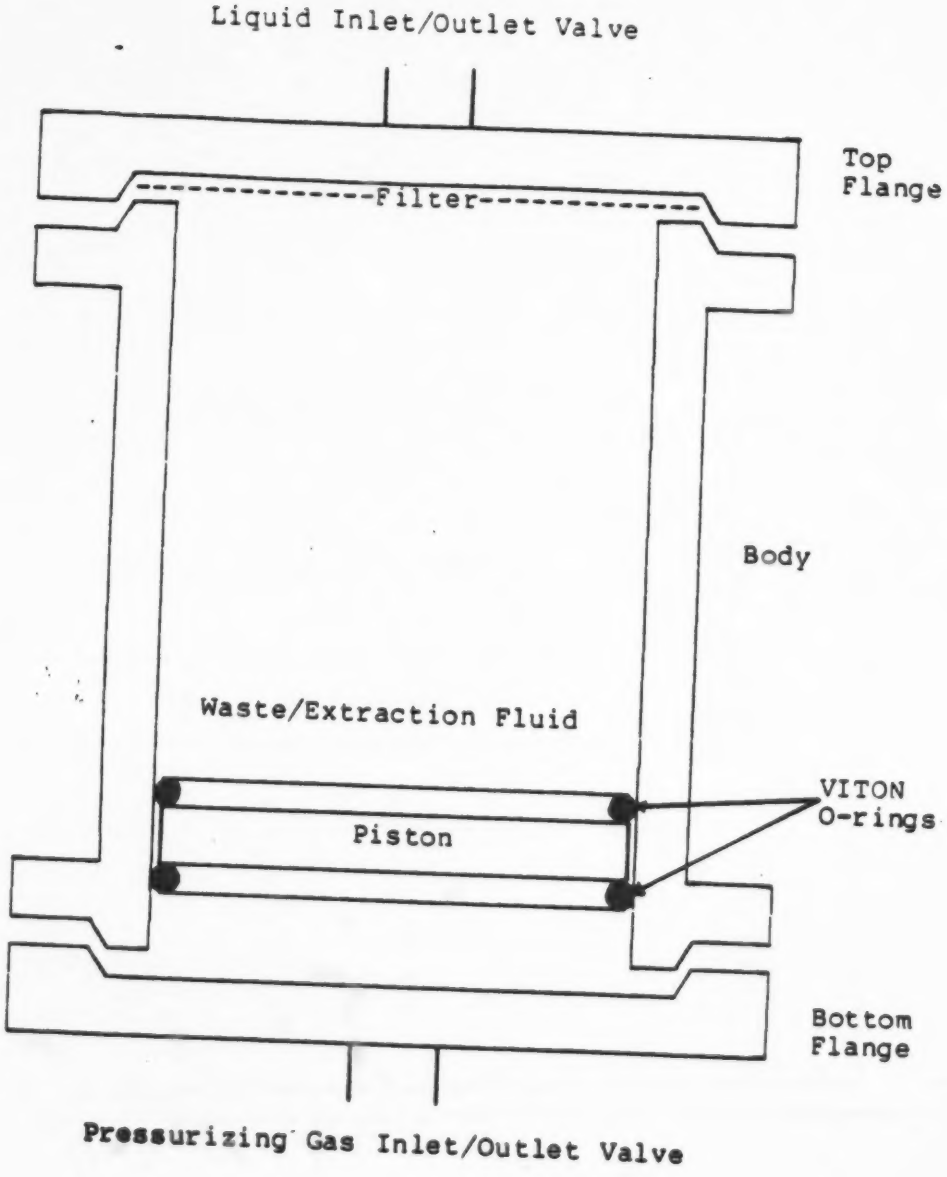


Figure 3: Zero-Headspace Extraction Vessel

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